TECHNICAL MEMORANDUM

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COPY: Mike Poore / CARB
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DATE: September 18, 2006
SUBJECT: CARB Laboratory Audit

Introduction

On June 20, 2006, a Technical Systems Audit (TSA) was conducted at the Northern Laboratories Branch of the California Air Resources Board (CARB) facilities located in Sacramento, California. The TSA was conducted as part of the US EPA's quality assurance oversight for the $PM_{2.5}$ Speciation Network. CARB has elected to use their own laboratory facilities to analyze many of the speciation samples collected within the state rather than use other laboratories which are available to perform this function under a federal contract.

This audit was performed by Steve Taylor and Jewell Smiley from EPA's National Air and Radiation Environmental Laboratory (NAREL) located in Montgomery, AL. Mathew Plate from EPA's Region 9 office located in San Francisco was also present for the audit. This TSA was a routine inspection of specific laboratory systems and operations at CARB that are required for the analysis of $PM_{2.5}$ Speciation samples. The last TSA performed by NAREL was conducted in March of 2004 [see reference 1].

Summary of Audit Proceedings

After a brief meeting with some of the CARB staff and supervisors, the audit team visited specific areas of the laboratory to interview those technical staff who actually perform the analyses. At least one member of the CARB staff was always available to escort and assist the auditors. The following specific areas at the CARB facilities were visited and inspected.

✓ Sample Receiving and Handling Laboratory - George Dunstan, Chris Barham

✓ Organic Carbon/Elemental Carbon (OC/EC) Laboratory - Peter Samra

✓ Gravimetric Laboratory - Debbie Moreno-Thornsberry

✓ X-Ray Fluorescence (XRF) Laboratory - Mike Humenny

✓ Ion Chromatography (IC) Laboratory - George Dunstan

Besides the areas mentioned above, interviews were also conducted with the following CARB staff.

✓ Michael Poore - Northern Laboratory Branch Chief

✓ Cliff Popejoy - Inorganics Laboratory Section Manager

✓ Samantha Scola - Air Pollution Specialist

✓ Dan Tackett - LIMS Specialist

CARB's Northern Laboratory Branch provides a large number of chemical analyses using many different analytical methods. However, this TSA focused exclusively on the techniques listed above which are used to analyze $PM_{2.5}$ filters collected at seven speciation sites and thirty mass sites. All seven of the speciation field sites use Met One SASS units for sample collection. CARB has been analyzing speciation samples since January of 2002.

The auditors were familiar with CARB's Quality Assurance Project Plan (QAPP) and pertinent SOPs. A few weeks before the TSA was scheduled, a set of single-blind Performance Evaluation (PE) samples were prepared at NAREL and submitted to CARB for analysis. Most of the results from these PE samples were available to discuss with CARB staff during the audit.

Sample Receiving and Handling Laboratory

George Dunstan and Chris Barham are immediately responsible for shipping clean filters to the field sites and receiving the loaded filters back at the lab. An SOP is available on the web that describes this critical process [see reference 2].

Sample receiving was the first area inspected, and all of the auditors were present to observe how samples were processed and handled. New clean filters are assembled into SASS canisters for shipment to the remote field sites. After the sampling event, the loaded filters are returned to the laboratory still mounted in the canister, but are cooled to approximately 4 °C for preservation during transit. Upon receipt at the laboratory, the canisters are removed from the shipping cooler, and the temperature is recorded. Each canister is disassembled, and the recovered filter is placed into a new container. The Nylon® filter is transferred to an extraction tube. The Teflon® and the quartz filters are transferred to Petri slides to await analysis. Canisters and filter holder cassettes are expensive and must be cleaned for reuse. A dishwasher was used to clean these items. Field blanks were used to monitor for accidental contamination of the filter media. A request was made to query the Laboratory Information Management System (LIMS) for the field blank results. A summary of those results is presented in the following table.

Table 1. Field Blank Results

Parameter	Instrument	Concentration (µg/filter) Average	Max	Min	Std. Dev.	LOD*	Number of Values
PM2.5 Mass	Balance	1.775	21	-21	7.264	1	173
Elemental Carbon	Carbon Anal.	0.006	0.230	0.000	0.029	9	173
Organic Carbon	Carbon Anal.	10.862	26.150	4.780	4.262	9	173
Ammonium	IC	0.270	0.760	0.070	0.146	0.5	173
Nitrate	IC	0.496	5.610	0.000	0.589	0.5	173
Potassium	IC	0.077	1.440	0.000	0.132	1	173
Sodium	IC	0.707	3.590	0.110	0.479	0.8	173
Sulfate	IC	0.041	0.440	0.000	0.087	2	173
Aluminum	XRF	0.167	1.250	-0.180	0.306	0.09	111
Antimony	XRF	0.020	0.210	-0.170	0.058	0.1	110
Arsenic	XRF	-0.004	0.040	-0.050	0.016	0.009	110
Barium	XRF	0.069	0.960	-0.540	0.187	0.05	111
Bromine	XRF	-0.001	0.020	-0.020	0.008	0.006	110
Calcium	XRF	0.028	0.490	-0.090	0.064	0.05	110
Chlorine	XRF	0.062	0.510	-0.300	0.128	0.03	110
Chromium	XRF	0.006	0.060	-0.010	0.011	0.02	110
Cobalt	XRF	0.004	0.010	-0.010	0.005	0.02	110
Copper	XRF	0.009	0.580	-0.050	0.058	0.01	110
Iron	XRF	0.020	0.210	-0.030	0.029	0.01	110
Lead	XRF	-0.021	0.060	-0.170	0.054	0.02	110
Manganese	XRF	0.002	0.020	-0.020	0.007	0.02	110
Mercury	XRF	0.008	0.060	-0.030	0.014	0.02	110
Molybdenum	XRF	0.001	0.050	-0.050	0.017	0.04	110
Nickel	XRF	0.002	0.010	-0.010	0.005	0.02	110
Phosphorus	XRF	0.044	0.320	-0.060	0.081	0.03	110
Potassium	XRF	0.034	0.130	-0.060	0.036	0.06	110
Rubidium	XRF	0.002	0.020	-0.020	0.009	0.007	110
Selenium	XRF	-0.001	0.020	-0.030	0.009	0.009	110
Silicon	XRF	0.101	0.620	-0.040	0.148	0.04	110
Strontium	XRF	0.004	0.020	-0.010	0.007	0.01	110
Sulfur	XRF	0.034	0.270	-0.030	0.061	0.03	110
Tin	XRF	0.007	0.190	-0.200	0.078	0.09	110
Titanium	XRF	0.009	0.100	-0.160	0.031	0.02	110
Vanadium	XRF	0.005	0.040	-0.060	0.013	0.02	110
Yttrium	XRF	0.006	0.030	-0.020	0.009	0.02	110
Zinc	XRF	0.007	0.230	-0.010	0.027	0.01	110
Zirconium	XRF	0.010	0.040	-0.010	0.012	0.02	82

**LOD = Limit of Detection*

The field blanks summarized in Table 1 were from the sampling period March 2002 to March 2006 except for most of the XRF parameters which were from March 2002 to August 2004. Only eighty-two values of zirconium are presented in Table 1 because the laboratory discontinued the analysis of zirconium after December of 2003.

A simple experiment was performed during the audit to measure the level of contamination that a filter may receive during canister assembly followed immediately by canister disassembly to retrieve the filter. Four sets of clean filters (four Teflon®, four Nylon®, and four quartz filters) were hand-carried from NAREL to the audit and were available for the experiment. Half of the filters (two sets) were assembled into "clean" canisters provided by CARB, and the remaining filters were treated as experimental control blanks since they were not removed from their containers during the TSA. Chris Barham is normally responsible for canister assembly and filter retrieval. Therefore, Chris performed the experiment during the audit. All of the filters were carried back to NAREL for analysis and the results are presented in Table 2.

Table 2. Results from Canister Assembly & Filter Retrieval Experiment

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
T06-11781	Teflon® test filter #1	PM2.5 Mass	Balance	-3
T06-11782	Teflon® test filter #2	PM2.5 Mass	Balance	-4
T06-11785	Teflon® control filter #1	PM2.5 Mass	Balance	-1
T06-11786	Teflon® control filter #2	PM2.5 Mass	Balance	0
Q06-11793	Quartz test filter #1	Elemental Carbon	Carbon Anal.	not detected
Q06-11794	Quartz test filter #2	Elemental Carbon	Carbon Anal.	not detected
Q06-11797	Quartz control filter #1	Elemental Carbon	Carbon Anal.	not detected
Q06-11798	Quartz control filter #2	Elemental Carbon	Carbon Anal.	not detected
Q06-11793	Quartz test filter #1	Organic Carbon	Carbon Anal.	3.9
Q06-11794	Quartz test filter #2	Organic Carbon	Carbon Anal.	3.3
Q06-11797	Quartz control filter #1	Organic Carbon	Carbon Anal.	5.7
Q06-11798	Quartz control filter #2	Organic Carbon	Carbon Anal.	3.4
N06-11787	Nylon® test filter #1	Nitrate	IC	0.44
N06-11788	Nylon® test filter #2	Nitrate	IC	0.78
N06-11791	Nylon® control filter #1	Nitrate	IC	0.72
N06-11792	Nylon® control filter #2	Nitrate	IC	0.47
N06-11787	Nylon® test filter #1	Sulfate	IC	not detected
N06-11788	Nylon® test filter #2	Sulfate	IC	not detected
N06-11791	Nylon® control filter #1	Sulfate	IC	not detected
N06-11792	Nylon® control filter #2	Sulfate	IC	not detected
N06-11787	Nylon® test filter #1	Ammonium	IC	not detected
N06-11788	Nylon® test filter #2	Ammonium	IC	not detected

Table 2. Results from Canister Assembly & Filter Retrieval Experiment

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
N06-11791	Nylon® control filter #1	Ammonium	IC	not detected
N06-11792	Nylon® control filter #2	Ammonium	IC	not detected
N06-11787	Nylon® test filter #1	Potassium	IC	not detected
N06-11788	Nylon® test filter #2	Potassium	IC	not detected
N06-11791	Nylon® control filter #1	Potassium	IC	not detected
N06-11792	Nylon® control filter #2	Potassium	IC	not detected
N06-11787	Nylon® test filter #1	Sodium	IC	not detected
N06-11788	Nylon® test filter #2	Sodium	IC	0.23
N06-11791	Nylon® control filter #1	Sodium	IC	not detected
N06-11792	Nylon® control filter #2	Sodium	IC	not detected

Results from the canister assembly experiment in Table 2 may be compared to the field blank results presented in Table 1. It is important to remember, however, that filters for the canister assembly experiment were supplied by NAREL, and results will be influenced by activities such as pre-cleaning the quartz and Nylon® filters at NAREL.

CARB maintains a stock of ready-to-go filters, and during the audit, a request was made to remove two sets of these clean filters from their stock. These stock filters were carried back to NAREL for analysis and the results are presented in Table 3.

Table 3. Results from Clean Filters Removed from CARB's Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
T06-11799	Teflon® test filter #1	PM2.5 Mass	Balance	2
T06-11800	Teflon® test filter #2	PM2.5 Mass	Balance	-2
Q06-11807	Quartz test filter #1	Elemental Carbon	Carbon Anal.	not detected
Q06-11808	Quartz test filter #2	Elemental Carbon	Carbon Anal.	not detected
Q06-11807	Quartz test filter #1	Organic Carbon	Carbon Anal.	3.8
Q06-11808	Quartz test filter #2	Organic Carbon	Carbon Anal.	4.1
N06-11803	Nylon® test filter #1	Nitrate	IC	0.80
N06-11804	Nylon® test filter #2	Nitrate	IC	1.00
N06-11803	Nylon® test filter #1	Sulfate	IC	not detected
N06-11804	Nylon® test filter #2	Sulfate	IC	not detected
N06-11803	Nylon® test filter #1	Ammonium	IC	not detected

Table 3. Results from Clean Filters Removed from CARB's Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
N06-11804	Nylon® test filter #2	Ammonium	IC	not detected
N06-11803	Nylon® test filter #1	Potassium	IC	not detected
N06-11804	Nylon® test filter #2	Potassium	IC	not detected
N06-11803	Nylon® test filter #1	Sodium	IC	not detected
N06-11804	Nylon® test filter #2	Sodium	IC	not detected

The results in Table 3 show that the quartz and Nylon® filters taken from CARB's stock were very clean. It should be explained that the Teflon® filters were evaluated by subtracting the tare mass determined at CARB from the tare mass determined several days later at NAREL. Table 3 shows very good agreement between CARB and NAREL for measuring the tare mass of the Teflon® filters. XRF analysis was not performed for the Teflon® filters listed in Table 2 and Table 3.

Good laboratory practices were generally observed for preparing the fresh canisters to send to the field and for retrieving the loaded filters following sample collection. No deficiencies were noted for this area of laboratory operations.

Organic Carbon/Elemental Carbon (OC/EC) Laboratory

The OC/EC analysis is normally performed by Peter Samra using an SOP that is available for viewing on the web (see reference 3). The carbon analyzer used at CARB is a DRI Model 2001 which is commercially available from Atmoslytic Inc. located in Calabasas, CA.

The analytical method currently used at CARB was implemented after NAREL's first TSA visit in the fall of 2002 (see reference 4). A problem was observed in the raw data thermograms at that time, which prompted CARB and NAREL to work together and develop a custom temperature protocol for CARB's instrument. CARB's custom heating protocol produced OC/EC results in good agreement with NAREL for samples that were split between the two labs. Table 4 shows the custom temperature protocol used at CARB along with two other popular protocols for comparison.

Table 4. Comparison of the Temperature Protocols for Three OC/EC Methods

CARB Method TOT Analysis	STN Method TOT Analysis	IMPROVE_A Method TOR Analysis	Carrier Gas
heater off (90s)	heater off (90s)	heater off (90s)	He Purge
250°C (180s)	310°C (60s)	140°C (150-580s)	He
400°C (150s)	480°C (60s)	279°C (150-580s)	He
550°C (150s)	615°C (60s)	480°C (150-580s)	He
700°C (270s)	900°C (90s)	580°C (150-580s)	He
heater off (60s)	heater off (40s)	-----	
550°C (100s)	600°C (35s)	580°C (150-580s)	He/O_2

Table 4. Comparison of the Temperature Protocols for Three OC/EC Methods

CARB Method TOT Analysis	STN Method TOT Analysis	IMPROVE_A Method TOR Analysis	Carrier Gas
650°C (100s)	675°C (45s)	740°C (150-580s)	He/O_2
750°C (100s)	750°C (45s)	840°C (150-580s)	He/O_2
850°C (100s)	825°C (45s)	-----	He/O_2
900°C (170s)	920°C (120s)	-----	He/O_2
heater off (200s)	heater off (110s)	heater off (200s)	He/O_2+IS

Heating protocols for the STN method and the IMPROVE_A method are also listed in Table 4. The STN method has been used at the Research Triangle Institute (RTI) for about six years since RTI was first awarded the national contract to analyze samples collected by the Speciation Trends Network (STN). The IMPROVE_A method has been used at the Desert Research Institute (DRI) for almost two years, and DRI is the national contract lab for the Interagency Monitoring of PROtected Visual Environments (IMPROVE) program. EPA has announced a plan to convert all of the STN sites, as well as the supplemental sites that CARB currently operates, to carbon sampling and analysis protocols that parallel the IMPROVE program (see reference 5).

NAREL has sponsored an inter-laboratory study for the past two years that includes replicate quartz filter sets distributed to CARB, DRI, and RTI. All of the participating labs were allowed to report OC/EC results from different instruments and also from different methods. CARB reported results for the past two years based upon its custom temperature protocol, and results from CARB were in good agreement with all of the other labs that used the STN method (see reference 6 for the 2005 study). For the more recent study, CARB also reported results using the IMPROVE_A method, and those results were in good agreement with other labs that used the IMPROVE_A method. CARB has decided to move ahead in preparing its OC/EC laboratory for the upcoming change to the IMPROVE_A method.

Raw data files were briefly discussed during the audit and examined later at NAREL. Some of the raw data thermograms will be included in the final report for the 2006 inter-laboratory study which should be released later this year. No new problems were observed for the OC/EC laboratory as a result of this audit.

Gravimetric Laboratory

Debbie Moreno-Thornsberry is the analyst primarily responsible for the gravimetric mass analysis following an SOP that is available for viewing on the web (see reference 7). The weighing lab is a dedicated room with controlled temperature, humidity, and dust. Chamber blanks which are left open inside the room are routinely analyzed to monitor dust. A Dickson data logger was brought to the audit and placed near CARB's device to measure temperature and humidity inside the weighing room. Good agreement was observed between the local device and the Dickson device.

The microbalance used to weigh the PM2.5 Teflon® filters was a Sartorius MC5. Even though excellent gravimetric mass results were reported for CARB's recent PE samples, four metallic mass

units were brought to the interview so that direct observations could be made as they were weighed. Results from the metallic mass units are presented in Table 4.

Table 5. Results from Weighing Metallic Units

Metallic weight ID	NAREL Value (mg)	CARB Value (mg)	Difference (mg)
MW06-11743	181.336	181.336	0.000
MW06-11744	88.206	88.206	0.000
MW06-11747	191.060	191.059	-0.001
MW06-11748	96.353	99.352	-0.001

No deficiencies for the gravimetric lab were noted. Overall good laboratory practices were observed during this TSA.

X-Ray Fluorescence (XRF) analysis Laboratory

The XRF analyses are currently performed by Mike Humenny, and his SOP is available on the web (see reference 8). Over the past several months Mike has been working with a new QuanX EC energy dispersive instrument available from the Thermo Electron Corporation. The new XRF instrument replaces a very old Kevex unit which was retired in 2003. The new instrument has been set up to acquire four spectra to support the analysis of each sample using instrument conditions that are listed in Table 6.

Table 6. XRF Analysis at the CARB Laboratory

Instrument: Thermo QuanX EC	Software: WinTrace 3.0.2			
	Instrument Conditions for Routine Sample Analysis			
Parameter	#1	#2	#3	#4
X-ray tube parameters:				
Tube voltage (kV)	10	30	50	50
Tube current (mA)	1.98	1.66	1.00	1.00
Tube anode material	rhodium	rhodium	rhodium	rhodium
Direct excitation of sample:				
Filter Material	cellulose	palladium	palladium	copper
Filter thickness (mm)	unknown	0.025 mm	0.125 mm	0.377 mm
Secondary excitation of sample:	none	None	None	None
Secondary Fluorescor				
Filter material				
Filter thickness (mm)				
Acquisition time (seconds)	800	400	400	800
Energy range acquired (keV)	0-10	0-20	0-40	0-40
Number of [MCA] channels	512	1024	2048	2048

Sample rotation (yes/no)	yes	yes	yes	yes
Beam spot size, diameter (mm)	unknown	unknown	unknown	unknown
Atmosphere (vacuum, He, air)	vacuum	vacuum	vacuum	vacuum
Elements Reported	Al Si P S Cl K Ca	Ti V Cr Mn Fe Co Ni Ba	Cu Zn As Se Br Rb Sr Y Mo Hg Pb	Sn Sb

The new instrument at CARB is very similar to the three XRF units at RTI which are also QuanX EC instruments with slightly different hardware options. It is worth noting that CARB has decided to report only twenty-eight elements while RTI reports forty-eight. Furthermore, CARB does not routinely report a dynamic uncertainty for each analytical result while all of the other speciation labs do this. CARB did have an estimated MDL for each element, however, which may be provided to the client upon request.

Most of the XRF field blank results summarized in Table 1 were not produced by the new instrument. The earliest field blanks were analyzed by CARB's old Kevex instrument until it was retired. Then a backlog of samples accumulated at CARB before the new instrument was received and ready for sample analysis. Some of the backlogged samples, including field blanks, were shipped to DRI for analysis, and therefore, only the most recent field blanks were analyzed by CARB's new QuanX EC instrument. Individual field blank results were provided during the audit and examined later at NAREL to look for trends over time. The most noticeable change in the field blank results over time was a sudden halt to reporting negative values after the old Kevex instrument was retired. This was true for all reported elements except aluminum.

The XRF lab was able to participate in NAREL's inter-laboratory study this year, and results from that study were discussed during the audit. Each participating lab analyzed a replicate set of six filters which had previously been analyzed at EPA's National Exposure Research Lab in Research Triangle Park, NC. CARB's results were in good agreement with the other participating labs except for the reported uncertainties. Many of CARB's uncertainties were smaller than those reported by other labs. CARB reported an uncertainty based exclusively upon the Poisson count statistics, and the other labs reported a more conservative uncertainty that included more sources of analytical error in the calculation. As stated earlier, the final report for the 2006 inter-laboratory study should be released later this year.

Ion Chromatography (IC) Laboratory

The IC analyses are performed by George Dunstan, and an SOP is available on the web that describes the IC analysis at CARB (see reference 9). The laboratory is equipped with an automated Dionex IC instrument. One channel is optimized for the analysis of anions and another channel is optimized for the analysis of cations. The lab also has access to equipment for cleaning and extracting Nylon® filters. Extractions are performed using an ultrasonic bath and a shaker table. Nanopure deionized water is the extraction solvent. Multilevel standards are used to develop calibration curves and establish retention times for the ions of interest. New calibration curves are checked against a standard from a secondary source. Fresh curves are prepared when the routine

check samples indicate excessive calibration drift. Replicate injections of low-level standards have been used to estimate sensitivity and low level precision. Duplicate injections of sample extracts have been used to evaluate mid-level precision. Blank spikes are extracted along with field samples to evaluate method accuracy. Statistically derived limits have been developed over time and are used to control the analytical system.

The only specific samples discussed were those from the recent PE study in which several laboratories analyzed a replicate set of single-blind filter samples. Records and raw data from the PE samples were examined during the audit. The results from the PE study indicate good performance from the IC laboratory.

The field blanks summarized in Table 1 shows respectably low levels of ion contamination. Therefore the overall process used to clean new Nylon® filters, assemble/disassemble canisters, and extract the filters offers an attractive baseline for IC measurements at CARB.

Other Staff Interviews

Mike Poore and Cliff Popejoy should be given much of the credit for making this TSA go smoothly. At least one of them was available every time the auditors needed information or assistance. Cliff was very helpful after the audit for providing NAREL with extra information as part of the audit follow-up.

Dan Tackett and Samantha Scola were helpful to provide the auditors with historical data that were requested during the audit. They provided the field blank data which was summarized in Table 1 of this report.

Conclusions

This audit was preceded by single-blind PE samples which were submitted to all of the labs that were inspected. Results from all of the PE samples were discussed with the analysts and supervisors involved. Some of the supporting raw data were examined during the audit, and some raw data were carried back to NAREL for examination as part of the audit follow-up. The auditors are pleased to report no significant negative findings. We do suggest, however, that the XRF results should be reported along with an uncertainty value, and the laboratory should decide which components of the overall uncertainty to include in the calculation.

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